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Technical News Bulletin

Porosity of Plated Coatings

BECAUSE the protective value of a plated coating on metal is dependent, to a large extent, on the exclusion of pores and other flaws in the coating, the Bureau has been investigating the causes, nature, and effect of porosity in plated coatings for the American Electroplaters' Society (AES). As part of this program, several different aspects of porosity have been studied under the direction of Fielding Ogburn and Donald R. Ernst. These include the development of an improved technique [1] to study the porosity of metallic electroplated coatings with a microscope, evaluation of the sensitivity of the ferroxyl test [2]—one of the generally accepted methods for detecting porosity—and determination of the influence of gas [3] on pore formation.

Further knowledge of these phases of electroplating and a satisfactory method of measuring the porosity should be of value both to the plating industry and to the science of electroplating. A continuous protective coating is particularly important in the field of aeronautics, where the metals used for jet engines and other high-temperature components must withstand great changes in temperature.

Microscope-Detection Method

Although much research has been directed toward pore detection and elimination, methods of examining pores on metallic coatings with microscopes have received little attention because most pores have diameters too small for adequate sectioning. Even if a pore is sectioned properly, the contours may be distorted by polishing and etching. So far, attempts to cut an entire pore parallel to its axis have been unsuccessful, so that a dot or oval is all that is seen.

"Parallel sectioning," a technique [1] developed by F. Ogburn, and D. W. Ernst, an AES Research Associate, involves polishing off some of the coating *perpendicular* to the axis of the pore and parallel to the basis metal. When the cut section is studied under a microscope, the pore will appear as a small dot which can easily be seen at 400× if the pore is larger than 0.05 mil in diameter. To make sure that the dot is not a surface defect introduced during polishing, successive layers are removed and examined until the underlying metal is reached. If the dot is a pore, it will reappear consistently at the same spot. A pantograph arrangement is used to relocate the exact position of the pore after successive removal of each layer.

With this technique, the size and shape of pores or other defects that are difficult to cross section can be determined and the damaging effects of atmospheric corrosion can be further studied.

Four types of pore defects were found in these studies: Pits (deadend pores), cavities (enclosed pores), continuous pores, and bridged-over pores. The relative frequency of these four types has not been determined.

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Sensitivity of Ferroxyl Test

Perhaps the best known porosity test for nickel coatings on steel is the ferroxyl test. Applying a solution of sodium chloride and potassium ferricyanide to the coating produces a blue spot wherever the basis metal is exposed. Because the reagent was found to attack the nickel coating and produce pores, this test has been modified [4] to eliminate any contact between the ferricyanide and nickel. However, since the overall sensitivity was not known, experiments [2] were conducted by the Bureau to determine the efficiency of the method.

Test specimens were prepared by plating steel sheets with gold on one side. The coating specimens were then tested with the modified ferroxyl test (solution of sodium chloride-potassium ferricyanide). Next the basis metal was dissolved by an acid solution, leaving the coating. This gold foil was photographed [5] by placing it on a photographic film and exposing to light. The resulting photographic record of the porosity, supplemented with measurements of pore size obtained by microscopic examination, were compared, spot by spot, with the pattern produced by the ferroxyl test.

From this experiment, it was found that the sensitivity of the ferroxyl test decreased as the pore size decreased. Virtually all pores 0.5 mil or more in diameter were detected by the ferroxyl test while very few of the pores less than 0.1 mil in diameter were detected. Since smaller pores are more numerous than larger pores, it is evident that the ferroxyl test has rather severe limitations.

Influence of Gas on Porosity

Several initiators of pore formation have been suggested. These are: Inclusions in the basis metal; surface contours and other discontinuities of the basis metal; foreign matter in the bath that settles on the cathode; and initial sites of nucleation on the basis metal which build up without filling all intervening spaces. No matter which of these—or other factors—initiates a pore, once it has been started subsequent deposition should bridge over it. The fact that this bridging over does not always continue with deposit build-up may be explained by gas discharge or bubble formation. From studies using parallel sectioning techniques, it seems likely that almost all pores follow the normal tendency to bridge over; those that are prevented from closing up because of gas accumulation give the deposit its resulting porosity.

One factor contributing to the formation of pores involves the growth of a deposit around a gas bubble. To study the effect of gas on porosity, a cell [3] was developed with a transparent bottom that facilitates observation of metal deposition. This cell has a concentric cylindrical electrode system so that the cathode (i.e., the metal being plated) is at the center of the system, pressed down on the thin cell bottom. Deposition forming around the periphery of the cathode can be observed through this transparent bottom. When the current density is increased until hydrogen evolves, and then decreased slightly, hydrogen bubbles remain attached to the plated surface. If the cell is operated at a low-current density, the deposit thickens at a uniform rate with few visible defects. At high current densities, hydrogen, evolved in great excess, produces a rough deposit that has poor adherence.

The relative rate of hydrogen discharge and metal deposition were found to determine whether a bubble is covered over by the deposit or enlarges itself. When a gas bubble is large enough to break away from the deposit, it leaves some gas in the pore formed by the deposit growing around the bubble. This gas effectively blocks the electrolyte from entering the pore and so prevents the deposit from forming across the pore opening. Although this experimental procedure differs from actual industrial plating conditions in several ways, observations seem to indicate that the pore growth mechanism it demonstrates is applicable on a broader scale.

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[1] Microscopic detection of porosity, *F. Ogburn, D. W. Ernst, Plating* **46,** *831 (1959).*

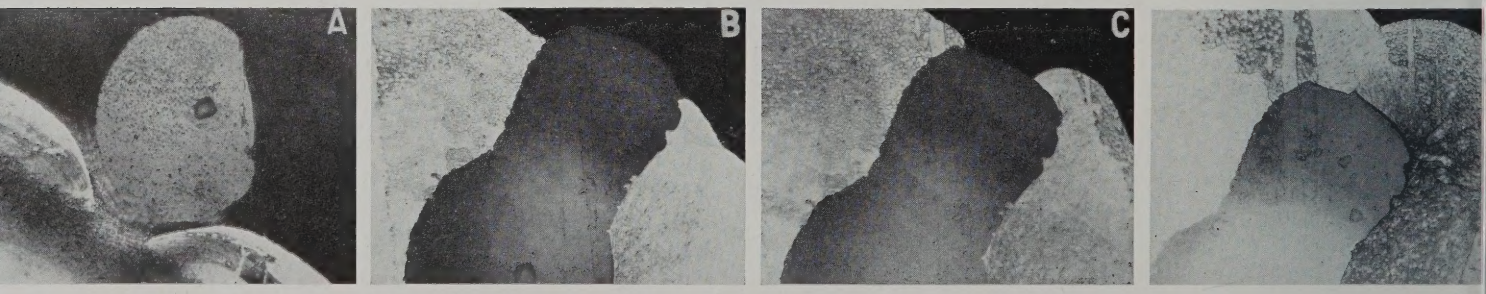
[2] An evaluation of the sensitivity of the ferroxyl test, *F. Ogburn, D. W. Ernst, W. H. Roberts, Plating (in press).*

[3] Influence of gas on porosity, *F. Ogburn, D. W. Ernst, Plating* **46,** 957 *(1959).*

[4] P. W. C. Strausser, *Proc. Am. Electroplaters' Soc.* **27,** *194 (1939).*

[5] The nature, cause and effect of the porosity in electrodeposits. II. Radiographic detection of porosity in electrodeposits, *F. Ogburn, M. Hilkert, Annual Tech. Proc. Am. Electroplaters' Soc. p. 256 (1956).* Porosity detection in plated coatings, *Tech. News Bul.* **41,** *44 (1957).*

Formation of pores due to gas bubble. Micrographs (100×) illustrate: (*A*) initiation of the bubble; (*B* and *C*) partial deposit growth around bubble; and (*D*) complete covering over.



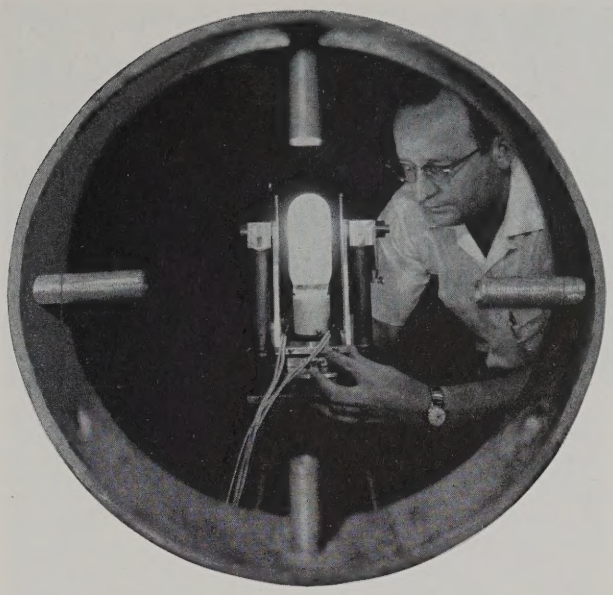
NEW CANDLEPOWER STANDARDS

NEW photometric standards for measuring the candlepower, or luminous intensity, of electric lamps are now available from the Bureau. They consist of 100-, 300-, and 500-w lamps with inside-frosted T-20 bulbs, C-13 monoplant filaments, and medium-bipost bases. These lamps are easily oriented on the photometric bar by means of the bipost bases, require no diaphragm when in use, follow closely the inverse square law, and are superior in the amount of candlepower variation they show with change in orientation to the clear-bulb lamp standards previously issued.

For many years coiled-filament clear-bulb lamps have been employed as reference standards for candlepower. However, the coiled filament has led to a problem of alinement in these lamps which has sometimes been difficult to overcome. In a recent study by L. E. Barbrow and S. W. Wilson of the photometry and colorimetry laboratory, variations in intensity up to 10 percent were noted when the clear-bulb lamps were tilted through an angle of 5 deg.[1]

In the experiments, if a lamp was oriented so that a maximum portion of the filament section at the rear of the coil showed between the front turns of the coil, maximum candlepower was obtained. When a lamp was positioned so that this rear section was eclipsed by the front turns, minimum candlepower resulted. Extreme caution was required in orienting the lamps about both the horizontal and vertical axes to achieve reproducible results.

To provide standards that could be more easily used by commercial laboratories, an inside-frosted lamp was designed with its filament, filament supports, and base posts in the same plane. Tests showed that because of the frosted bulb, little or no variation in candlepower occurs with change of lamp orientation, either in tilt or rotation about the lamp axis. In addition, it was found that the lamps follow the inverse square law to within 1.0 percent for distances of 50 cm or greater between lamp and photometer, and to well within 0.5 percent for distances of 1 m or greater. These distances are measured from the plane of the base pins to the photometer.

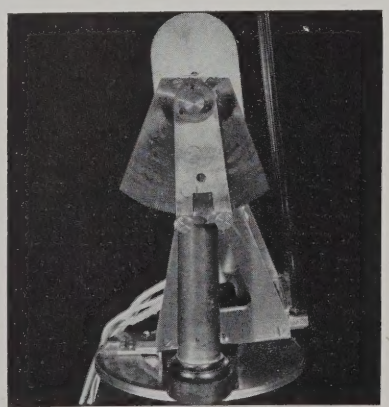


S. W. Wilson adjusts one of the new 100-w lamp standards on the photometric bar for calibration. A barrier-layer cell photometer at the far end of the bar is shielded by baffles.

A study was made of the effect of filament displacement from the center of the bulb. Two lamps were fabricated with the filament displaced 1 cm from the plane of the center of the base posts, which was approximately 1 cm from the center of the lamp bulb. The study showed the inverse square law was closely followed if distances were measured to the plane of the filament, and not to the center of the bulb.

Accordingly, in calibrating the inside-frosted standards at the Bureau, the distance from photometer to lamp is measured to the center of the filament supports near the base posts (where they are visible through a clear portion of the bulb), or to the center of the base posts, or to the center of the base-post socket holes. With any of these procedures the distance may be accurately measured to the filament plane.

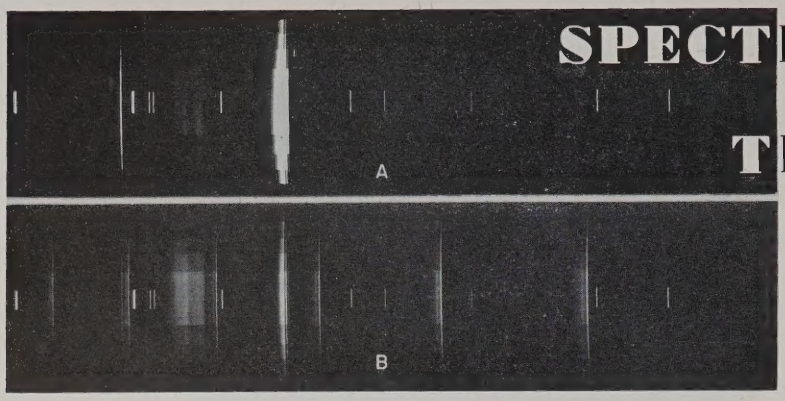
The frosted-bulb standards may be procured from the Photometry and Colorimetry Section, National Bureau of Standards, Washington 25, D.C. They are priced at $37.00 for the 100-w size; $34.00 for the 300-w size; and $35.00 for the 500-w size.



There is no appreciable change in candlepower even when the frosted bulb lamp standard is tilted through a 5-deg angle as shown here.

[1] *For further technical details, see* Vertical distribution of light from gas-filled candlepower standards, *by L. E. Barbrow and S. W. Wilson, Illum. Eng.* **53,** *645 (1958).*

SPECTROSCOPIC STUDIES of TRAPPED RADICALS

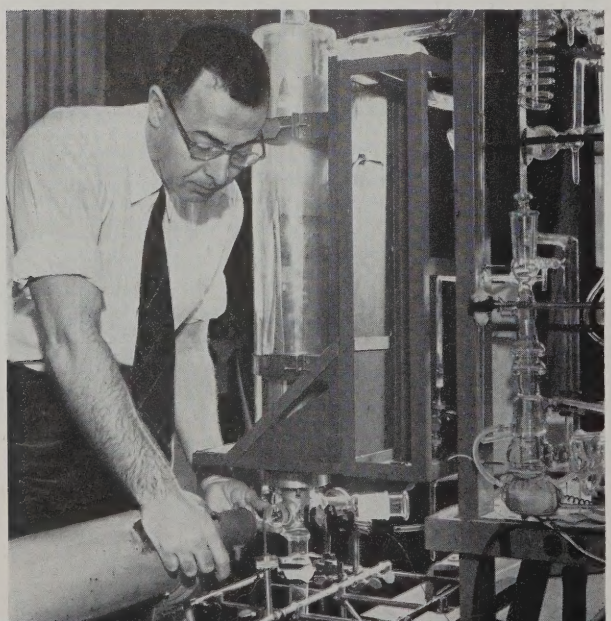


Spectra obtained in the electron bombardment of nitrogen condensed on a metal surface at 4°K. *A* shows a pure nitrogen spectrum. *B* shows effect of addition of 1 percent oxygen. Not only does this small amount of oxygen introduce additional bands but it greatly reduces the intensity of the primary transition. Short lines indicate calibration spectrum.

TO PROVIDE basic data on the properties and behavior of stabilized free radicals, the Bureau, under the sponsorship of the Department of Defense, has made an extensive spectroscopic study of highly reactive atoms and excited molecules trapped in solids at low temperature.

A number of methods are available for producing such electronically excited molecules and atoms in solids at low temperatures. The Bureau employed primarily two techniques in order to present a consistent overall picture of observations. In one aspect of the work, M. Peyron, guest research worker from the University of Lyon, collaborated with H. P. Broida and H. W. Brown of the Free Radicals Section in investigating reactive fragments condensed from nitrogen gas passed through an electric discharge.[1] In the other phase of the study, carried out by E. Hörl of the electron physics laboratory, nitrogen atoms were produced by the electron bombardment of nitrogen condensed on a cold surface.[2] Analogous studies with oxygen were made by L. Schoen and H. P. Broida.

Apparatus used in studies of the light emitted by oxygen-rare gas deposits bombarded by electrons. L. J. Schoen focuses a spectroscope on the cold surface where free radicals are trapped. These reactive fragments are produced when electrons impinge on the condensate.



Free radicals have been known to exist for about 30 yr but only lately have they become the object of widespread interest. The Bureau is now engaged in a 3-yr program of free-radical studies.[3] In most of this work, free radicals have been produced by the discharge technique mentioned above.[4] The glow phenomena observed here were found to be quite similar to those observed in early work where free radicals were produced by electron bombardment. This led the Bureau to repeat both experiments to provide a complete set of data on radical behavior.

The apparatus used in studying the products of a gas discharge is quite simple in principle. Gaseous nitrogen flows through a quartz tube in which an electrodeless microwave discharge is maintained by a waveguide. The products from the discharge are condensed on a block cooled by liquid helium or hydrogen. The light emitted by these discharge products emerges through quartz windows and is analzed by glass- and quartz-prism and grating spectrographs and an infrared spectrometer.

In studying free radicals generated by electron bombardment, nitrogen is first condensed on a metal surface cooled to 4° K by means of a liquid helium bath. This surface is then used as an electron target, and in the bombardment free radicals are produced. Windows allow the glow phenomena to be observed visually and with spectrographs. Glass prism spectrographs, quartz prism instruments, and a transmission grating instrument were used.

Light emission, which can be attributed to nitrogen atoms and molecules, is observed in three distinct periods in each of the studies. First, there is the glow during deposition or bombardment. This is immediately followed by an afterglow when excitation has ceased. Later a warmup glow appears as the coolant evaporates and radicals are released.

The main features of the spectra obtained by the two methods are very similar.[5] This agreement leads to the conclusion that only certain excited states are capable of radiative transitions.

Through spectral observations of the emitted light it has been possible to separate most of the features of the solid glows into atomic and molecular transitions and to identify many of the emitters. An interpretation of one of the strong spectral features indicates that emission is due to a loosely bonded complex of N and N_2.[6] With this model it is possible to characterize the observed emission as simple combinations of electronic transitions of the nitrogen atoms and of vibrational transitions of the N_2 molecules. For example, one particular line group observed in both studies cor-

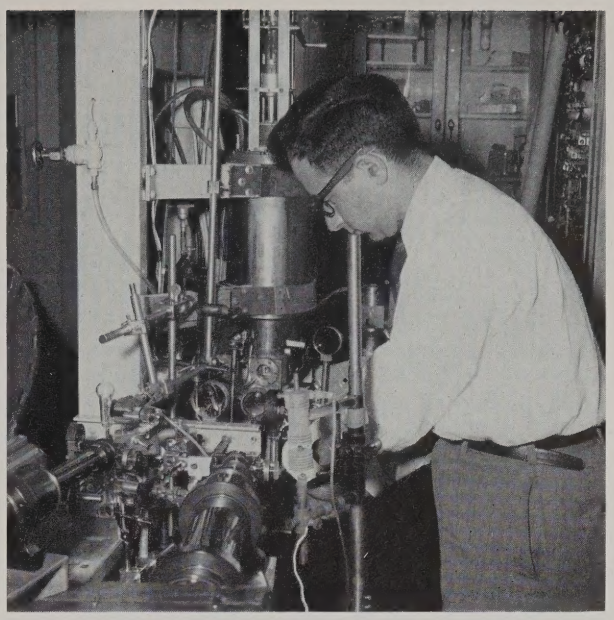
responds to an identifiable transition of a trapped atom. An associated line group seems to correspond to the same transition with the N_2 molecule in a vibrationally excited state.

Under special conditions, spectral lines appear which are attributed to one of the transitions of atomic *oxygen*. In this case, oxygen atoms are thought to be trapped in a weakly bonded N_2–O complex.

However, this picture of a loosely bonded complex, so effective in explaining atomic lines, does not apply in interpreting some other observations, such as the molecular features strengthened or introduced when impurities are added to the nitrogen. A pure nitrogen spectrum consists mainly of atomic features (lines) which are not significantly affected by the addition of other gases such as oxygen and argon. On the other hand, such components have a very definite effect on molecular features (bands), increasing the intensity of some and introducing still others. For example, argon in large amounts greatly increases the intensity of the Vegard-Kaplan bands which were first observed and identified in the initial production of nitrogen free radicals 30 yr ago.[7] Under other conditions two sets of bands which can be traced to the NO molecule appear. In nitrogen containing hydrogen or water vapor, bands appear which are interpreted as transitions of the NH radical.

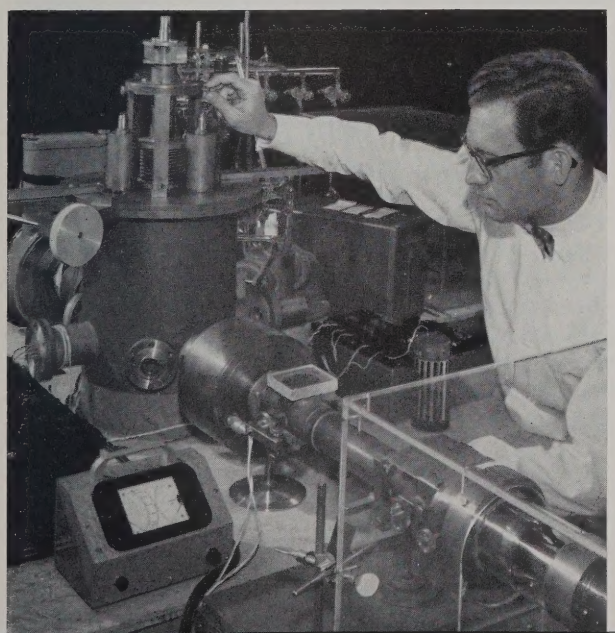
The emission spectra from an electron-bombarded deposit of a rare gas and oxygen were studied between 1.1° and 20°K. This analysis provides information about the interaction between the reactive species and the solid matrix. For example, wavelength measurements of a particular band series (the Herzberg bands) in oxygen show a distinct change in the electronic term, indicated by a change in the position of the band system for the electronic transition in the solid relative to that in the gas. Changes in vibrational frequencies and anharmonicities in the solid during such transitions appear to be relatively small and subject to some degree of uncertainty at present. Precise knowledge of these latter characteristics is limited by the width of spectra originating in the condensed phase.

Through these spectroscopic studies a large amount of information concerning free radicals at low temperatures has been obtained. However, many effects are left unexplained so that further comprehensive experimental studies are required. It is hoped that future research will provide a basis for an acceptable theory of the interactions that the emitting species undergoes with the solid or with neighboring species, which will lead to a quantitative understanding of the properties and structure of solids containing free radicals.



A. M. Bass adjusts apparatus for producing free radicals at temperatures near absolute zero by a discharge method. Metal cylinder (center) contains liquid helium in thermal contact with cold surfaces on which frozen free radicals are deposited. The light emitted by the solids thus deposited can be observed through the quartz window below the cylinder and is analyzed on the spectroscopes in the left foreground.

Apparatus used in studies of trapped radicals produced by the electron bombardment of nitrogen condensates. Electrons from the electron gun (right foreground) impinge on the helium-cooled, nitrogen-coated target inside the cylindrical cryostat. E. Hörl adjusts the position of this target.



[1] Spectra emitted from solid nitrogen condensed at very low temperatures from a gas discharge, *by M. Peyron and H. P. Broida, J. Chem. Phys.* **30,** *139 (1959).*

[2] Light emission from solid nitrogen during and after electron bombardment, *by E. M. Hőrl, J. Molecular Spectroscopy (in press).*

[3] Free radicals research program, *NBS Tech. News Bul.* **41,** *1 (1957).*

[4] Low-temperature storage of free radicals, *NBS Tech. News Bul.* **40,** *112 (1956).*

[5] Spectroscopic evidence for triatomic nitrogen in solids at very low temperature, *by M. Peyron, E. M. Hőrl, H. W. Brown, and H. P. Broida, J. Chem. Phys.* **30,** *1304 (1959).*

[6] Theory of the forbidden transition of nitrogen atoms trapped in solids, *by C. M. Herzfeld, Phys. Rev.* **107,** *1239 (1957).*

[7] The auroral spectrum and the upper atmosphere, *by L. Vegard, Nature* **113,** *716 (1924).*

Vibration Pickup Calibration Service Established

THE Bureau has recently established a vibration pickup calibration service [1] for the range of 10 to 2,000 cps at accelerations up to 10 g. Previously available on a limited basis only,[2] this service will provide industrial and Government laboratories with a standard against which their pickups may be compared. These calibrated pickups can in turn be used to standardize pickups that are needed to test missiles, rockets, aircraft, and other structures.

In this service, the errors in motion applied to pickups do not exceed 1 percent up to 900 cps and 2 percent between 900 and 2,000 cps. When a pickup is supplied without an indicator or recorder, the Bureau determines the magnitude and phase angle of the calibration factor. Calibrations are made at 3 different accelerations at 10 different frequencies, for a total of 30 calibration points.[3] Dynamic calibrations are also made at frequencies from 10 to 15 cps at double displacement amplitudes up to 0.5 in.

Dynamic calibrations between 2,000 and 20,000 cps are also performed at the Bureau, but with different equipment. These calibrations are usually made by the fringe disappearance method.[5] In this method, the displacement amplitude usually corresponds to one fringe disappearance, approximately 4 μ in.

The calibration service is a result of the Bureau's continuing program of developing vibration standards. In this program, a vibration standard consisting of a commercial electrodynamic exciter calibrated by the reciprocity method [4] was used to provide a limited calibration service. This standard was thoroughly tested and was found to require modification before it could be used in a routine calibration service.

Electrodynamic Standard

The vibration standard has three moving parts: A table on which pickups are rigidly mounted; a driving coil that produces vibration of the moving parts; and a velocity-sensing coil that is used to measure the velocity of the mounting table. These parts are joined by a shaft and they vibrate together as a unit. The vibrating unit is suspended from a frame by leaf springs. A large field magnet and a small permanent magnet are also attached to the frame. The attachment of the permanent magnet is made with soft springs. The driving coil fits within the large field magnet, and the velocity-sensing coil fits into an annular gap in the small permanent magnet.

The large field magnet is excited when direct current passes through its field coil. An alternating current is sent through the driving coil, causing a reaction between its field and the field of the large magnet. This reaction gives an axial vibratory motion to the moving parts. As the mounting table vibrates, the velocity-sensing coil moves with it. This coil moves in the field of the small permanent magnet that remains stationary, and the voltage generated in it is therefore proportional to the absolute velocity of the table and pickup. The outputs of the pickup and sensing coil are then compared to determine the calibration factor of the pickup. Because the standard is mounted on trunnions, calibrations can be made with the standard and pickup in either the vertical or horizontal position.

The standard is usually operated at frequencies above the natural frequency of the system composed of the springs and moving parts. At these frequencies, the table has an acceleration waveform similar to the waveform of the current in the driving coil. However, distortion in the motion is introduced due to undesirable resonances in the standard. It is important that the standard apply pure sinusoidal axial motion. It was for this purpose that the standard was modified.

R. Bouche records calibration data from vibration pickup mounted on the Bureau's newly modified vibration standard (left). At each of three rectangular blocks, helical coil springs are attached to wires to minimize transverse motion during vibration.

Modification of the Standard

The major change in the standard is the addition of a tensioned wire system. In this system, three wires are attached to one end of the moving parts, and three others are attached to the other end of the same parts. Each set of three wires is arranged in a plane perpendicular to the axial motion of the vibrator, and each wire is 120 deg from each of the others in its set. The outer ends of all wires are attached to the frame

by helical coil springs. The attachment is made so that the tension load in each wire can be adjusted easily to compensate for the static deflection of the moving parts when a pickup is attached to the mounting table that is operating in a horizontal plane.

If the springs deflect appreciably from the weight of the moving parts, the coils are recentered on their respective magnets. The coils must be centered in the axial and transverse direction. They can easily be centered within $\frac{1}{32}$ in., and greater off-centering can be tolerated before there is significant change in the sensitivity of the velocity-sensing coil.

Significant transverse motions have been eliminated by the use of this wire system. Results of testing several vibration standards showed that with conventional leaf springs, there was significant transverse motion at many vibration frequencies.

Music wires of 0.018- and 0.037-in. diam were used, with about equal success in eliminating transverse motion. Changes in the tension in the wires, however, affect their resonance response.

When the tension in the vertical wires was about 9 lb, and the side wire 8 lb, fundamental bending-mode resonances were found at 400 and 430 cps. For the vertical wires, second-mode resonances were found near 890 cps, nearly twice the fundamental resonances. There was no observable effect of the second-mode resonances in the side wires, nor of the higher-mode resonances in any of the wires.

When the tension loads were reduced to 1 and 2 lb in the side and vertical wires, respectively, there was only slight transverse motion in the standard at 530 and 950 cps due to bending-mode resonances of the moving parts, and there was no transverse motion caused by the resonant response of the wires.

When wires were used, there was little transverse motion. The Bureau was able, therefore, to expand its services from a limited basis to a routine calibration service. To make certain there is no change in the standard, it is calibrated yearly by the reciprocity method.

First- and second-mode axial resonances of the moving parts of the standard were found to occur near 2,100 and 4,500 cps, depending on the mechanical impedance of the pickup. If excessive harmonic distortion is present in the power amplifier driving the standard, these resonances would be excited when calibrating at subharmonic frequencies, and some distortion would occur in the axial motion. This condition has not been a serious problem in the standard.

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[1] *For further technical details, see* Improved standard for the calibration of vibration pickups, *by R. R. Bouche, Soc. for Exp. Stress Anal. (May 1959).*

[2] Electrodynamic standards for vibration pickups, *NBS Tech. News Bul.* **41,** *11 (1957).*

[3] Test Fee Schedules of the National Bureau of Standards—Mechanics, *reprinted from Federal Register* **28,** *188 (1958). Available without charge from the National Bureau of Standards, U.S. Department of Commerce, Washington 25, D.C.*

[4] Calibration of pickups by the reciprocity method, *by S. Levy and R. R. Bouche, J. Research NBS* **57,** *227 (1956) RP2714.*

[5] Some developments in vibration measurement, *by S. Edelman, E. Jones, and E. R. Smith, J. Acous. Soc. Am.* **27,** *728 (1955).*

NEW PUBLICATION:

Testing of Glass Volumetric Apparatus

IN ORDER to encourage high quality in the manufacture and accuracy in the use of precision glass apparatus for measuring volume, the National Bureau of Standards has formulated the specifications contained in this publication. Methods of calibrating and using such equipment are described, as well as the tests performed by the Bureau and procedures for submitting instruments for test. The information applies to burets, pipets, and other precision-grade apparatus.

The Bureau admits for test only apparatus conforming to the standards listed in the circular, since apparatus of lower quality is not sufficiently precise to justify the labor required for its accurate calibration. It is therefore to the advantage of users of such apparatus, if they wish to avail themselves of the Bureau's services, to insist on conformity with the standard specification.

Testing of Glass Volumetric Apparatus, National Bureau of Standards Circular 602, by J. C. Hughes, 14 pages, issued April 1, 1959, 20 cents. Supersedes Circular 434. (Order from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C.)

dielectric

TO PROVIDE a better understanding of the properties exhibited by dielectric materials, the Bureau is conducting a program of fundamental research sponsored in part by the Department of Defense. Primary objectives of this program are to explain observed dielectric phenomena and to develop theories which can be used to predict the behavior of certain materials. In most of the work, a solid state approach is employed to probe the molecular behavior responsible for dielectric phenomena. These studies are complemented by efforts to improve the accuracy of dielectric measurements in the fields of electricity, electromagnetic waves, electronics, solid state research, polymer studies, and physical chemistry.

During the past few years, demands for dielectric materials designed for specific applications have become more and more insistent. In the missile and aircraft fields, for example, dielectrics with electrical properties that are relatively independent of temperature are needed. To create dielectrics for special purposes in this way, the effects of various physical conditions on electrical properties must be determined, and the relationship between molecular behavior and electrical properties must also be established. Programs of dielectric studies, carried out by the Bureau's Dielectrics Section [1] and ferroelectricity group, located in Washington, and the Radio and Microwave Materials Section of the Boulder (Colo.) Laboratories, provide concepts for such "molecular engineering".

Basic Dielectric Studies

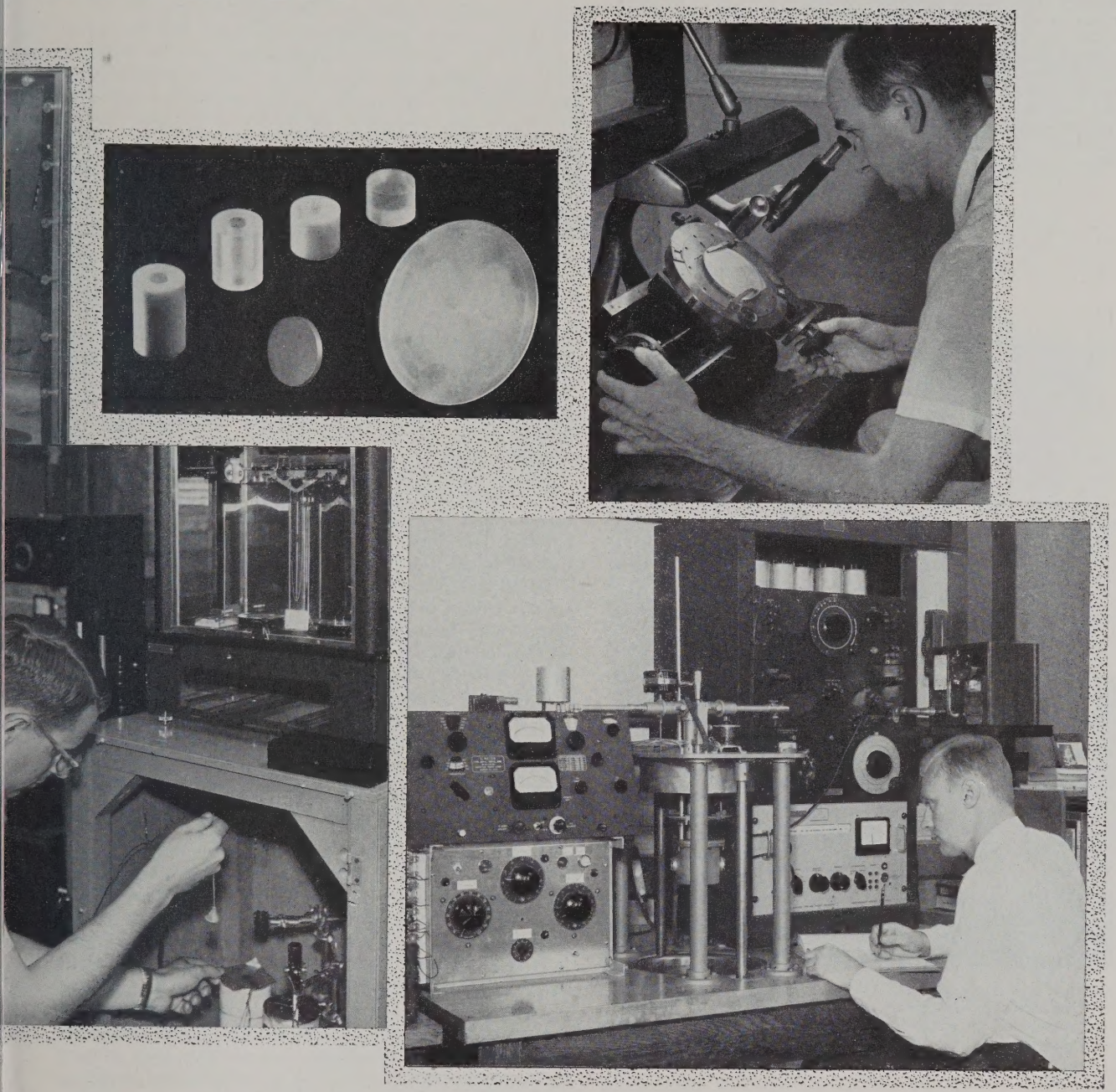
Many dielectric phenomena are associated with a property known as "electric polarization". Part of this electric polarization is due to a displacement of the bound charges in molecules when a voltage is applied. Little or no dielectric loss is associated with this contribution. Electric polarization can also take the form of interfacial polarization, that is, polarization arising from movement of electric charges on the interface between two discrete phases. Still other electric polarization arises from the presence of orientable permanent electric dipoles in molecules. Loss caused by "frictional" restraint on the motions of the ions or dipoles is associated with these last two types of polarization.

The Dielectrics Section is interested primarily in dipole polarization because it provides a basis for studying both intermolecular and intramolecular motions in such important materials as high polymers and for establishing the relationships which exist between the electrical characteristics and certain chemical and physical characteristics. Dipole polarization also is a factor in the dielectric spectrum through the radiofrequency range. It is involved in the nonresonant microwave spectroscopy of gases carried on by the Radio and Microwave Materials Section to investigate induced polarization during collision processes.

When the loss index (loss factor) of a material containing dipoles is plotted against the logarithm of the applied frequency, a maximum caused by a relaxation mechanism occurs at some particular frequency value. The height of this maximum, the frequency at which it occurs, the breadth of the loss peak, and the change of these parameters with temperature, provide information which leads to a fundamental understanding of many of the important electrical properties of the material. Such parameters can also be correlated with other properties, for example, mechanical relaxation.

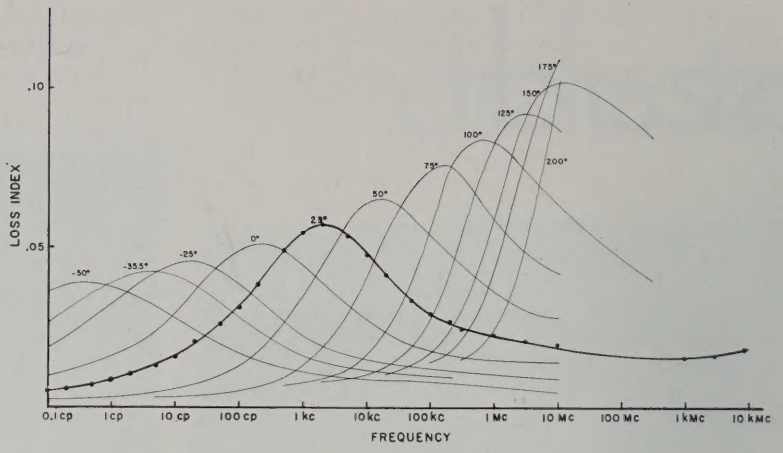
Above left: **A. H. Scott introduces a sample into the humidity cabinet, used to determine the effect of atmospheric conditions on dielectrics. A rubber glove, like the one on the left side of the box, will seal the opening.** ***Above center:*** **Group of specimens typical of those studied. Small disk, electrode-plated poly(methyl methacrylate); large disk, polystyrene; opaque rods (first and third from left), polychlorotrifluoroethylene; and clear rods, polystyrene.** ***Above right:*** **W. P. Harris uses traveling microscope to measure the dimensions of a dielectric sample under consideration as a reference standard.** ***Below left:*** **Studying the crystallization of dielectrics. Since the specific volume of a specimen is affected by crystallinity, J. J. Weeks weighs the sample in silicone oil—a medium of known density.** ***Below right:*** **A. J. Curtis uses a special micrometer electrode holder designed for use from the liquid-nitrogen range up to 300°C. To achieve very low temperatures, holder (center) is placed in a Dewar containing liquid nitrogen. The top of the Dewar, which can be raised up to the holder, can be seen at table level. At left is a modified commercial resonant circuit and to the right of the holder is a capacitance bridge.**

research



To make measurements over a wide range of frequencies and temperatures, a wide variety of special bridges, circuits, holders, transmission lines, resonant cavities, and cells have been put into use. Dielectric constants and dissipation factors are measured at both the Washington Laboratories and the Boulder Laboratories over a frequency range of 0.1 to 10^{10} cps at temperatures from $-100°$ to $+500°$ C. In addition, facilities for millimeter-wave work are available at Boulder, and studies have been made at the low-frequency end of the dielectric spectrum involving frequencies whose periods are days in length.

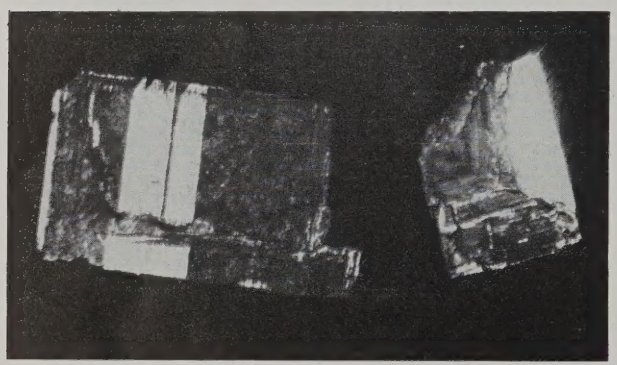
Just as frequency determines which dielectric measuring technique must be used, temperature dictates the



Dielectric loss index versus frequency for polychlorotrifluoroethylene at selected temperatures from −50° to 200° C. The degree of crystallinity in these specimens was 80 percent. For each temperature value, there is a frequency at which the loss factor reaches a peak. As this frequency increases with temperature, the height of the peak also increases.

structure of dielectric cells. The Bureau has developed several systems. Among these are dielectric cells with micrometer-electrode holders for use at temperatures up to 500° C and down to −196° C.[2] In such holders, two electrodes, one movable and one stationary, contact the specimen, and a precision micrometer determines the position of the movable electrode.

To study materials that pass from liquid form to solid form in the temperature range of interest, a "fusion cell" has been designed. This is similar to the other holders but allows the material in liquid phase to be held in the space between and around the electrodes. It applies pressure to the movable electrode as the sample is solidifying, thus preventing voids



Barium titanate crystals used in the study of conductivity and instability in ferroelectric materials. The crystals are observed with polarized light to show domain formations.

from forming as the material contracts. Work is presently underway to determine the effects of solidification on the electrical properties of materials such as 2,2-dinitropropane, an interesting nearly spherical molecule that undergoes rotation in the solid state.

To study materials as they make a transition from gaseous dielectric behavior to liquid dielectric behavior, resonant cavities for compressed gases have been developed.[3] Studies made with these show that liquid-like behavior can set in at pressures of less than an atmosphere for the strong dipolar gases.[4] Further work is being initiated which will determine dielectric losses in very-low-loss gases, as, for example, sulfur hexafluoride, which very closely approximates a spherical molecule.

All of this experimental work is supported by an active theoretical program. Some of the theoretical topics under study are dipole relaxation in molecular crystals, the dielectric properties of semicrystalline polymers, and the nature of the glassy state.

In the study of crystalline nonmetallic inorganic solids, carried on by the ferroelectricity group and the Radio and Microwave Materials Section, measuring circuits similar to those mentioned are employed. These materials, however, present somewhat different problems, both theoretical and empirical. Whereas with polymeric materials it is desirable to consider the polarization in terms of molecular structure, here crystal structure is of utmost importance. Contributions to the dielectric constant arise from electron displacement, local displacements of the nuclei forming the lattice, and migrations of these nuclei to other sites. These contributions will be affected not only by the crystal structure and orientation but also by crystalline defects such as impurities, vacancies, and domain boundaries.

A detailed study in the Boulder Laboratories of polarization and conductivity in barium titanate [5] has led to formulations which take into account the presence of free charges near the domain walls. These charges lower the wall energy so that there is a difference between calculated and measured coercive force. The new equations explain variations in hysteresis loop shape, the dependence of conductivity on polarization, and the variation of switching time with various parameters. To extend this work, single crystals of barium titanate with various conductivities are needed. It is anticipated that controlled conductivities may be achieved by bombarding crystals with electrons and measuring the change in shape of the hysteresis loop as the free electron content of the crystals changes.

One major difference in experimental techinque arises from the high dielectric constant of the titanate

materials presently being studied. Instead of employing the micrometer holders used in the other studies, electrodes must be directly bonded to the surface of the specimen. The electrodes are made to cover the entire surface to minimize edge effects.

As ceramic materials are especially sensitive to temperature and humidity fluctuations, studies must be carried out in a controlled environment. A constant-temperature holder has therefore been developed for use with a Schering bridge. This holder consists of an inner chamber in which 19 specimens can be simultaneously maintained at 35.1°C by the circulating water from a constant-temperature bath. Between the inner chamber and an insulating outer chamber, a flow of dry nitrogen eliminates humidity effects. A second specimen holder designed for studies of the effect of temperature variation on individual specimens has also been constructed. It will be used from room temperature up to 200°C.

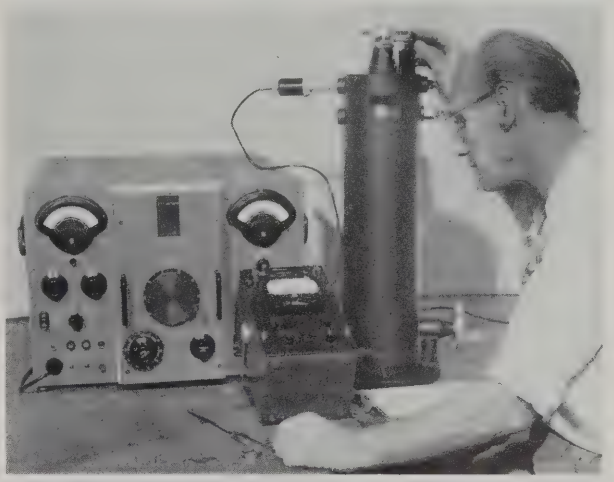
An added difficulty encountered in making accurate dielectric measurements on ceramics arises from the large effects of even small changes in specimen fabrication. To compensate for possible error, considerable attention is given to improving measurment techniques and characterizing specimens. Two efforts are being made in this direction. First, an exploratory program on the effects of the electrodes has resulted in the adoption of indium-gallium amalgam instead of silver as the accepted electrode material. Second, some single crystals to supplement ceramic specimens are being produced with the idea that the crystals will be easier to characterize.

Through these continuous efforts to improve accuracy and reliability, it is hoped that eventually the complete relationship between the composition and electrical characteristics of ceramics can be worked out. For the present, the ferroelectricity group will continue to concentrate on the titanates in general, and on barium titanate in particular. With its aim to provide a better understanding of the dielectric behavior of a single group of interesting materials, this program forms an integral part of the dielectric studies throughout the Bureau.

While ceramics are crystalline by definition, many polymers are neither completely crystalline nor completely amorphous. Therefore, to obtain a comprehensive picture of these materials, it is necessary to determine the degree of crystallinity as a function of such variables as temperature and previous history. The degree of crystallinity is related in a simple manner to the density of the polymer. This density is easily measured by weighing the material in silicone oil. As the density of the oil at any temperature can be measured, a means for studying the amount and speed of crystallization in the specimen at various temperatures is thus provided. A comprehensive set of measurements on the important plastic polychlorotrifluoroethylene at temperatures from –50° to 250°C has provided such data on the relationship of specific volume to degree of crystallinity.[6] Dielectric measurements have been made over approximately the same range of temperature, and correlated with the degree of crystallinity. Similar dielectric measurements are now being made on nylon at temperatures from –100° to 175° C. These measurements are complicated by the appearance of a large d-c conductance at the higher temperatures, and by an electret effect.[7]

Theoretical studies, which are being conducted to explain the relationship between the electrical properties, the amount of crystallinity, and the effects of temperature, will form a prominent part of the dielectrics program on semicrystalline polymers for some time in the future.

Certain crystals such as sapphire, certain molecules such as the noble gases (helium, argon, etc.), and some other molecules, have extremely low loss which can conveniently be studied at microwave frequencies. Also measurements on very pure aluminum oxide reveal



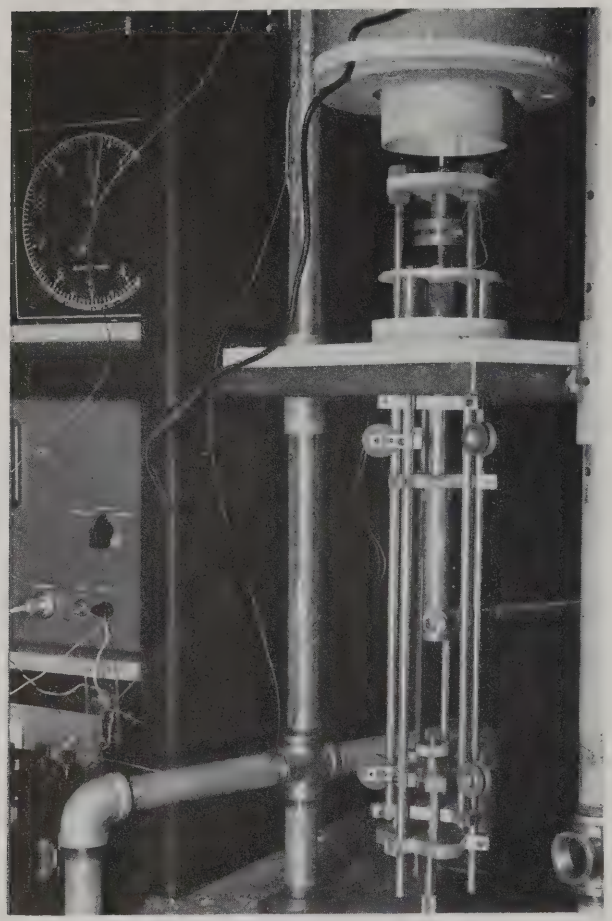
E. C. Bamberger, of the Bureau's Boulder (Colo.) laboratories, makes dielectric measurements using the 100- to 500-Mc reentrant variable-length cavity resonator.

dissipation factors smaller than most materials now known. This demonstrates the possibility of obtaining even lower losses with very pure substances. As the theory of these losses is incomplete, the Radio and Microwave Materials Section is conducting theoretical studies and making more accurate new measurements. Such investigations will have practical applications especially important at higher temperatures where low-loss materials are essentially nonexistent at present. Further work in these areas is dependent upon obtaining pure materials under carefully controlled conditions.

In the interaction between electromagnetic waves and matter, conductivity properties of materials are essentially inseparable from the dielectric properties. At radio and microwave frequencies, conductivity studies are therefore an important component of any dielectrics program. In this connection, the Radio Materials and Microwave Section is studying the tensor conductivity of semiconductors such as single crystals

of germanium at microwave frequencies under different physical conditions. Some important questions in the theory of solid state will be investigated in this work. It is expected that these studies will yield a better understanding of the crystal lattice forces and processes. The results may prove of value in microwave engineering if the tensor properties for radio waves are appreciable.

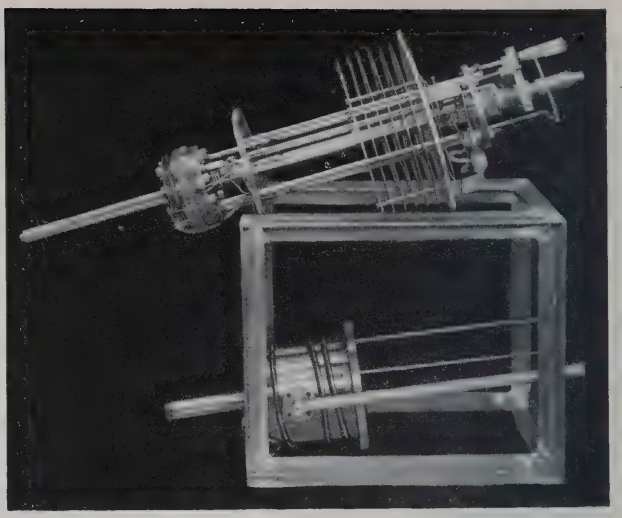
Among other examples of the Bureau's research in dielectrics are refractometer studies. The refractometer [8] determines the dielectric properties of the atmosphere, which in turn determine the propagation



Dielectric specimen holder developed by the Bureau for use at temperatures up to 500° C. Controls for the electrode spacing and measurement of the position of the movable electrode are located outside the heated space to eliminate the effects of such temperatures. Holder is constructed of stainless steel.

characteristics of the troposphere, so important to radar and other means of microwave communication. The Bureau's work constitutes the background for successful applications in this field for military and other purposes.

Propagation of electromagnetic waves or radio signals through a material is governed by the product of



Disassembled fusion cell. The principal unit consists of the cell itself and the controls. Below is a unit which houses the cell.

the material's magnetic permeability and its dielectric constant. For many substances the magnetic permeability is unity. In such cases the travel of waves in the material is dependent only on the complex dielectric constant or the ability of the material to "store" electrical energy. The Radio and Microwave Materials Section has participated in studies on the dielectric properties of various propagation media, such as ferrites, soils, tar sands, and oil-bearing sandstones. Ferrites are magnetic materials that have important magnetic properties at radio and microwave frequencies and have gyrotropic or directional characteristics important in microwave applications. They also have very high dielectric constants which are currently being investigated. Studies on soils are important in locating radio transmitters and antennas and establishing and predicting radio communication links. Tar sands and oil-bearing sandstones contain valuable oil deposits which do not flow under their normal temperature environment in which the tar sands and paraffin in oil are solid. By the application of suitable radio or microwave energy it is hoped to tap these resources by warming the deposits sufficiently for flow. Here the properties of composite dielectrics are of particular interest.

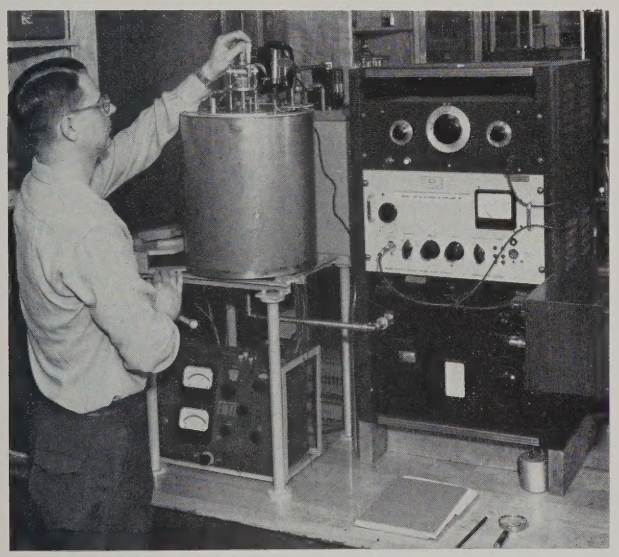
Standardizing Dielectric Measurements

Accuracy in dielectric measurements is basic to all electrical applications, where dielectrics often set limits on voltage, frequency, absorption, reflection, and temperature of operation. For this reason, the electrical properties of dielectrics must be determined before the materials are incorporated in electrical equipment such as capacitors, transmission lines, antennas, transmitters, transducers, and generators. Furthermore, as small

changes in production techniques may radically alter the electrical properties of the material, additional measurements are sometimes necessary to control the quality of finished equipment.

The Bureau helps to meet the ever-increasing need for accuracy and reliability by improving measuring techniques and developing reference standards.[9] This work is carried out principally by the Dielectrics Section in the Washington Laboratories and the Radio and Microwave Materials Section in the Boulder Laboratories. The Boulder Laboratories are responsible for dielectric tests and standards at and above 30 kc, and the Washington Laboratories are responsible for similar work at lower frequencies. In addition, results obtained by the Bureau's chemistry laboratories in research on molecular dynamics are frequently applicable to standardization procedures.

The Bureau calibrates the dielectric properties of transfer standard samples submitted by other laboratories, but would prefer to issue its own standard samples of selected material having known dielectric behavior as a function of environmental conditions and



J. J. Weeks makes a micrometer reading in determining the dielectric properties of a material undergoing a change of state. A special fusion cell is used for this work, either with the bridge shown at right or the higher-frequency resonant circuit located beneath the fusion cell housing.

time. Such reference standards may then be used with confidence by industry and other institutions in calibrating any dielectric measuring system. Careful studies of materials for reference standards are therefore being conducted in both of the standards laboratories, with support from the chemistry laboratories.

Such a standard may be a gas, liquid, or solid. Recommended values of the dielectric constants of a selected number of liquids and gases that are useful as

Growing single crystals of inorganic nonmetallic materials. Powdered material drops through flame, melts, and forms a crystal on the vertical rod at the base of the furnace. After closing the furnace, Katherine King will continue to check on the process, using an optical filter to look through a slit in the furnace wall.

standards are therefore included in Bureau tabulations of dielectric properties.[10] These tables also provide a concise and, wherever feasible, critically evaluated listing of dielectric data on a wide variety of substances. One table gives the dielectric constants for over 800 organic and inorganic liquids as a function of temperature; another presents the electric dipole moments, derived from microwave spectroscopy and measurements of the dielectric constants, for about 350 gases; and a third lists values of the dielectric constant and dielectric loss index as a function of frequency for about 200 liquids and a number of dilute aqueous and nonaqueous solutions.

Several factors enter into the reliability of a solid as a dielectric standard. First of all, for a material to be suitable as a standard it must be uniform. The Bureau therefore studies the uniformity of dielectric properties over a single specimen by making measurements with different-sized electrodes. Uniformity between specimens is, of course, determined by measuring several specimens.

Another important consideration in selecting a material as a dielectric standard is the influence of environmental factors on the stability of electrical properties. To determine the effect of one of the most important of these factors—relative humidity—the Bureau measures specimens after they have been kept for several weeks in a desiccator where the relative humid-

ity is less than 1 percent, and again after they have been kept for several weeks in a relative humidity of 50 percent. The most promising materials are those whose electrical properties are not greatly affected by the humidity. Humidity studies are presently underway on poly(methyl methacrylate), polystyrene, polyethylene, and fused quartz to determine their suitability as dielectric standards.

In further efforts to assure accuracy, the Bureau is cooperating with the American Society for Testing Materials in developing improved techniques for two-terminal measurements and for dielectric measurements of microwave ferrites and ceramics.

Two types of two-terminal measurements are in use: the open-type measurement, where edge and ground capacitance corrections are applied, and the micrometer-electrode holder measurement, which eliminates these corrections. A disadvantage inherent in the latter technique is the requirement that the specimen be a flat plate with sides perpendicular to the surface, and edges which are sharp with no rounding or chipping. The Bureau is presently studying micrometer-electrode holder techniques in an effort to improve this type of measurement. Accuracy of the dielectric constant measurement by this technique is affected by the means used to set the electrodes and to measure the spacing between them, while accuracy of the dissipation factor measurement is affected by changes in relative humidity. For the measurement of high-loss materials, the loss in the holder is a negligible correction, but for low-loss materials it can be appreciable—in some cases it is of the same order of magnitude as the loss in the specimen.

At microwave frequencies exact solutions for a cavity resonator with a dielectric (or magnetic) sample have been developed and programed on the automatic calculator. Certain errors, such as those due to frequency pulling by the coupling devices and the hole for inserting the sample, have been evaluated in this way. An automatic dielectrometer, covering a frequency range of 500 kc to 800 Mc and temperature range from –40° to 350°C, is now being developed.

Accuracy in dielectric measurements at low frequencies is dependent not only on measuring techniques and devices, but ultimately on the accuracy of the National standard of capacitance as maintained by the Bureau. At present, a new improved absolute standard, developed last year, is coming into use.[11] This standard, accurate to a few parts in a million, was made possible by a special 3-winding transformer bridge and a 3-terminal assembly whose capacitance depends solely on its length.[12] Although the present status of dielectric measurements and the lack of reproducibility among samples of the same dielectric material do not allow the accuracy of the standard to be fully utilized in dielectric research, the bridge itself is finding immediate use. In a modified form, which will sacrifice some accuracy for a wide frequency range, the transformer bridge will be used to measure dielectric losses.

At radio and microwave frequencies, the accuracy of dielectric measurement depends ultimately on the basic radio and microwave standards, which are calculable geometrical structures. A recent development here is the construction of an impedance standard calculated mathematically and measured experimentally with agreement to better than 0.1 percent.[13] The ability to measure impedance accurately then leads to accurate dielectric measurements. Equally valuable are the exact solutions for cavity geometries with perturbation corrections for all departures from an ideal structure. An example of such a departure is a coupling device or a hole for inserting the sample, as mentioned above.

Through this comprehensive program of dielectric activities, which begins with the development of a primary standard, extends through improved measurements, and results in materials data and an understanding of fundamental behavior, the Bureau provides the basis for the technological application of dielectric materials. Efforts will continue along the lines described, with emphasis continually shifting toward basic studies as the accuracies necessary for the effective application of data are realized.

---

[1] Dielectrics Section established, *NBS Tech. News Bul.* **41,** *15 (1957).*

[2] Versatile specimen holder for dielectric measurements, *NBS Tech. News Bul.* **41,** *14 (1957).*

[3] Cavity resonators for dielectric spectroscopy of compressed gases, *by H. E. Bussey and G. Birnbaum, Rev. Sci. Instr. (in press for Oct. 1959).*

[4] Change in the inversion spectrum of ND_3 from resonant to nonresonant absorption, *by G. Birnbaum and A. A. Maryott, Phys. Rev.* **92,** *270 (1953).*

[5] Conductivity and instabilities of barium titanate, *by R. C. Powell, Proceedings Special Technical Conference on Solid State Dielectric and Magnetic Devices, Catholic University of America (April 1957).*

[6] Specific volume and degree of semicrystalline polychlorotrifluoroethylene, and estimated specific volumes of the pure amorphous and crystalline phases, *by J. D. Hoffman and J. J. Weeks, J. Research NBS* **60,** *465 (1958) RP2862.*

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[8] A recording microwave refractometer, *G. Birnbaum, Rev. Sci. Instr.* **21** *(1950). See also,* Measurement of variations in atmospheric refractive index with an airborne microwave refractometer, *by H. E. Bussey and G. Birnbaum, J. Research NBS* **51,** *171 (1953).*

[9] R–F dielectric standards, *NBS Tech. News Bul.* **35,** *77 (1951).*

[10] Determination of dielectric constants of pure liquids, *NBS Tech. News Bul.* **43,** *81 (1959);* Dielectric properties of pure substances, *NBS Tech. News Bul.* **37,** *158 (1953);* *NBS Circ. 514,* Table of dielectric constants of pure liquids; *NBS Circ. 537,* Table of dielectric constants and electric dipole moments of substances in the gaseous state; *NBS Circ. 589,* Tables of dielectric dispersion data for pure liquids and dilute solutions. *Circulars available from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. (Prices 30 cents, 20 cents, and 50 cents, respectively).*

[11] New capacitance standards, *NBS Tech. News Bul.* **42,** *229 (1958).*

[12] A new theorem in electrostatics and its application to calculable standards of capacitance, *by A. M. Thompson and D. G. Lampard, Nature* **177,** *888 (1956).*

[13] Recently developed microwave impedance standards and methods of measurement, *by R. W. Beatty and D. M. Kerns, IRE Trans. Instr.* **I–7,** *319, 1958.*

Patent for Free-Radical Stabilization

A PATENT [1] has recently been granted to the Bureau for its original high-temperature discharge, low-temperature collection technique for producing and stabilizing free radicals.[2] This method was developed in 1954 by staff members H. P. Broida and J. R. Pellam.[3] Their work allows unstable atoms and free radicals, known to exist but momentarily in flames and hot gases, to be produced in an electric discharge, frozen into immobility, and trapped in solid form. Because these atoms are frozen in the excited state, they can be conveniently studied by optical spectroscopy.

This procedure forms the basis for much of the Free Radicals Research Program presently under way at the Bureau.[4] The program provides an integrated approach to studies of trapped radicals and the substances which contain them. Through the understanding of radical behavior provided by such basic research, the potentialities of the reactive fragments for probing atomic and molecular behavior are being realized.

The technique for free-radical stabilization has been employed by the Bureau in studies of nitrogen, oxygen, and other gases.[5] One study of the chemical processes taking place in oxygen resulted in a highly efficient method for producing ozone.[6] Industry is also making use of the very important tool provided by the radical trapping technique, especially in the field of low-temperature chemistry.

[1] *United States Patent No. 2,892,766 of June 30, 1959.*
[2] Low-temperature storage of free radicals, *NBS Tech. News Bul.* **40,** *112 (1956).*
[3] *Now on the faculty of the California Institute of Technology.*
[4] Free Radicals Research Program, *NBS Tech. News Bul.* **41,** *1 (1957).*
[5] Spectroscopic studies of trapped radicals, *NBS Tech. News Bul.* *(see p. 164).*
[6] Low temperature production of ozone, *NBS Tech. News Bul.* **43,** *29 (1959).*

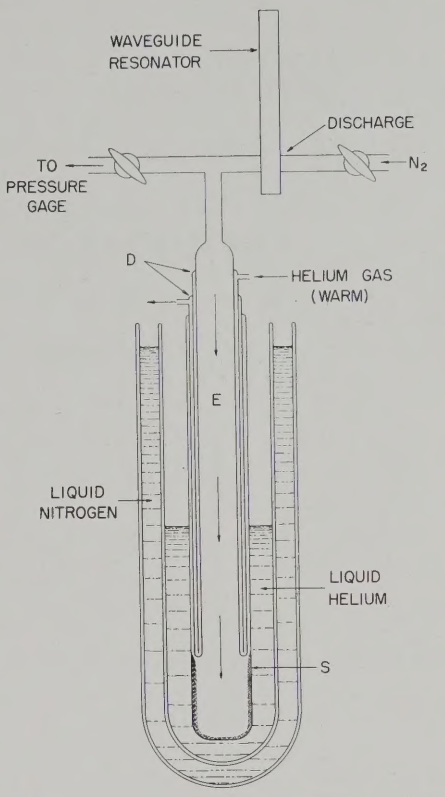

Diagram of the recently patented apparatus developed by the Bureau for collecting and storing free radicals at very low temperature. Nitrogen or other gas is admitted through an electrodeless discharge in the waveguide resonator, where it is broken up into free radicals. The surface S, immersed in liquid helium, acts as a trap, freezing out all gases except helium among the discharge products at temperatures above 4.2° K. To prevent solidification of the discharge products at temperatures above 4.2° K, warm helium gas is passed between the compound walls of the tube E. The liquid nitrogen in the outer flask helps to insulate the liquid helium.

Publications of the National Bureau of Standards

Periodicals

Journal of Research of the National Bureau of Standards

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic $4.00; foreign $4.75.

Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic $2.25; foreign $2.75.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic $2.25; foreign $2.75.

Section D. Radio Propagation. Issued six times a year. Annual subscription: Domestic $4.00; foreign $4.75.

Section D. *Radio Propagation, Volume* **63D,** *No. 2, September-October 1959. Approx. issue date September 10, 1959.*

Stratification in the lower ionosphere, C. Ellyett and J. F. Watts.

Effect of small irregularities on the constitutive relations for the ionosphere, K. C. Budden.

Ionospheric investigations using the sweep-frequency pulse technique at oblique incidence, V. Agy and K. Davis.

Fields in electrically short ground systems: an experimental study, A. N. Smith and T. E. Devaney.

Diffraction of electromagnetic waves by smooth obstacles for grazing angles, J. R. Wait and A. M. Conda.

Very-low-frequency radiation spectra of lightning discharges, W. L. Taylor and A. G. Jean.

Radio-wave scattering by tropospheric irregularities, A. D. Wheelon.

Study at 1046 megacycles per second of the reflection coefficient of irregular terrain at grazing angles, R. E. McGavin and L. J. Maloney.

Synoptic study of the vertical distribution of the radio refractive index, B. R. Bean, L. P. Riggs, and J. D. Horn.

Technical News Bulletin, Volume **45,** No. 7, July 1959 and No. 8, August 1959. 15 cents. Annual subscription $1.50. 75 cents additional for foreign mailing.

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NBS Publications (Continued)

Basic Radio Propagation Predictions for October 1959. Three months in advance. CRPL–D179. Issued July 1959. 10 cents. Annual subscription $1.00. 50 cents additional for foreign mailing.

Technical Notes

Technical Notes are available only from the Office of Technical Services, U.S. Department of Commerce, Washington 25, D.C. (Order by PB number.)

Thermodynamic properties of helium at low temperatures and high pressures, D. B. Mann and R. B. Stewart. TN8 (PB151367). $1.25.

Transmission loss in radio propagation—II, K. A. Norton. TN12 (PB151371). $3.00.

Technical considerations leading to an optimum allocation of radio frequencies in the band 25 to 60 mc, K. A. Norton. TN13 (PB151372). $2.50.

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Variations of surface tension with body and surface concentrations. A. B. Bestul. J. Am. Ceram. Soc. **42**, 5, 236 (1959).

On the internal resistance of dry cells. A new pulse method. Ralph J. Brodd. J. Electrochem. Soc. **106**, 6, 471 (1959).

Aerodynamic heating and fatigue. Wilhelmina D. Kroll. NASA Memo 6–4–59W. National Aeronautics and Space Admin., Washington, D.C. (1959).

Patents

(The following United States Patents have been granted to NBS inventors during the quarter ending June 30, 1959, and have been assigned to the U.S. Government as represented by the Secretary of the Department noted in parentheses.)

2,879,491 March 24, 1959. Plug system for joining electric subassembly to chassis. Gustave Shapiro (Navy).

2,882,038 April 14, 1959. Large-capacity balance. Henry H. Russell (Commerce).

2,882,501 April 14, 1959. Micropotentiometer. Myron C. Selby (Commerce).

2,882,694 April 21, 1959. Cool-down apparatus for cryogenic liquid containers. Peter C. Vander Arend and Douglas Mann (Navy).

2,882,822 April 21, 1959. Fuze housing. Clarence B. Crane (Navy).

2,883,527 April 21, 1959. Stabilized nonlinear amplifiers. Gail E. Boggs (Commerce).

2,883,620 April 21, 1959. High frequency power measuring bridge circuit. Myron C. Selby, Charles M. Allred, Paul A. Hudson, and Ira S. Berry (Commerce).

2,886,621 May 12, 1959. Sequential filled reserve battery. Wilbur S. Hinman, Jr. (Army).

2,887,622 May 19, 1959. Electrical circuit pattern tester. Charles C. Rayburn and Robert L. Henry (Navy).

2,889,493 June 2, 1959. Miniature radar assembly. Robert K-F Scal and Clinton O. Lindseth (Navy).

2,889,519 June 2, 1959. Clamp-type current transducer. Carroll Stansbury and George Franklin Montgomery (Commerce).

2,890,863 June 16, 1959. Combined pressure cooling system and chassis for miniaturized radar. Robert K-F Scal (Navy).

2,892,093 June 23, 1959. Fuze. Joseph E. Henderson (Navy).

2,892,129 June 23, 1959. Electronic module mounting device. Robert L. Henry Jr. (Navy).

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